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THE EFFECT OF OXYGEN
CONCENTRATION ON THE BOUNDARY
LUBRICATING CHARACTERISTICS OF
AN UNFORMULATED C-ETHER TO 300° C

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THE EFFECT OF OXYGEN CONCENTRATION ON THE BOUNDARY LUBRICATING CHARACTERISTICS OF AN UNFORMULATED C-ETHER TO 300° C

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SUMMARY

The effect of oxygen concentration on the boundary lubricating characteristics of an unformulated C-ether (modified polyphenyl ether) was studied with the use of a ball-on-disk sliding-friction apparatus. Experiments were performed in atmospheres with oxygen concentrations of 20 percent (air) and 0.001 percent (nitrogen) and with five intermediate concentrations. Results were compared with those obtained with a five-ring polyphenyl ether in two atmospheres (air and nitrogen).

Other experimental conditions included a load of 1 kilogram (initial Hertz stress of 10^9 N/m²), a sliding speed of 17 meters per minute (100 rpm), disk temperatures ranging from 25° to 300° C (77° to 572° F), and a test duration of 25 minutes. Test specimens were made of CVM (consumable electrode vacuum melted) M-50 steel.

The C-ether yielded lower wear than did a five-ring polyphenyl ether at a 20-percent oxygen level (air) from 50° to 195° C (122° to 383° F) and at a 0.001-percent oxygen level (nitrogen) from 50° to 265° C (122° to 509° F). Higher wear was observed with the C-ether from 195° to 300° C (383° to 572° F) in air and from 265° to 300° C (509° to 572° F) in nitrogen.

The C-ether exhibited lower wear at high oxygen levels (10 to 20 percent O₂) from 25° to 200° C (77° to 392° F) and at low oxygen levels (0.001 to 1.0 percent O₂) from 200° to 300° C (392° to 572° F).

In general, the coefficient of friction for the C-ether increased with increasing temperature and decreasing oxygen concentration. The C-ether yielded either similar or lower coefficients of friction than did the five-ring polyphenyl ether in air and in nitrogen.

Friction polymer was observed around the wear scars of most test specimens. Friction polymer generation and its effect on wear is discussed in light of current theories.

INTRODUCTION

Advanced aircraft and reentry vehicles will place increased thermal stresses on hydraulic fluids and lubricants. Maximum fluid temperatures in excess of 316°C (600°F) have been estimated for future applications (refs. 1 to 5). At these elevated temperatures, fluids must operate without appreciable degradation and also must provide effective lubrication for bearings and hydraulic-system components.

The C-ethers, which are structurally related to the polyphenyl ethers, are a promising class of fluids for possible high-temperature applications (refs. 6 and 7). They have excellent thermal stability (thermal decomposition temperature of 390°C (734°F) as measured with an isoteniscope), good oxidation stability to 260°C (500°F), and lower pour points (-29°C ; -20°F) than polyphenyl ether (5°C ; 40°F). They also exhibit low vapor pressure (low volatility), high surface tension (less starvation prone), and high bulk moduli.

The main deficiencies of the C-ethers have been their poor boundary lubricating ability and poor wetting characteristics (refs. 8 and 9). Heat-transfer (cooling) problems have been encountered with this fluid class (ref. 2) and are probably a result of its poor wetting properties.

The C-ethers, polyphenyl ethers, and other aromatic fluids have yielded poorer boundary lubricating characteristics in inert atmospheres (nitrogen or argon) than in air (refs. 8, 10, 11, and 12). This is in direct contrast to nonaromatic hydrocarbons, which yield lower wear in the absence of oxygen under conditions less severe than those required for seizure (ref. 13).

The objectives of this investigation were (1) to determine the effect of various oxygen concentrations, varying from 20 percent (air) to 0.001 percent (nitrogen), on the boundary lubricating characteristics of an unformulated C-ether from 25° to 300°C (77° to 572°F) and (2) to compare these results with some obtained with a five-ring polyphenyl ether in air and in nitrogen.

Experiments were conducted with a ball-on-disk sliding-friction apparatus. Test conditions included a load of 1 kilogram (initial Hertz stress of 10^9 N/m^2), a sliding speed of 17 meters per minute (100 rpm), and a test duration of 25 minutes. Test specimens were made of CVM (consumable electrode vacuum melted) M-50 steel.

EXPERIMENTAL LUBRICANTS

The C-ether fluid used in these studies is a blend of three- and four-ring components that are structurally similar to the polyphenyl ethers. This is a base fluid and contains only an antifoam additive. The polyphenyl ether used in these studies is an

TABLE I. - TYPICAL PROPERTIES OF THE EXPERIMENTAL FLUIDS

[Manufacturer's data except where noted otherwise.]

Property	Five-ring poly-phenyl ether	C-ether
Kinematic viscosity, m^2/sec (cS), at		
38° C (100° F)	3.6×10^{-4} (360)	2.5×10^{-5} (25)
99° C (210° F)	1.3×10^{-5} (13)	4.1×10^{-6} (4.1)
300° C (572° F)	9.5×10^{-7} (0.95)	6.9×10^{-7} (0.69)
Pour point, °C (°F)	5 (40)	-29 (-20)
Flash point, °C (°F)	288 (550)	230 (445)
Fire point, °C (°F)	350 (662)	285 (540)
Density at 38° C (100° F), kg/m^3 (g/ml)	1.19 (1.19×10^3)	1.18 (1.18×10^3)
Thermal decomposition (isoteniscope), °C (°F)	443 (830)	390 (734)
Vapor pressure at 371° C (700° F), torr	28	140
Surface tension at 23° C (73° F), dynes/cm (N/cm)	$^{a}46.0$ (4.6×10^{-4})	$^{a}44.8$ (4.48×10^{-4})

^aData from ref. 22.

isomeric mixture of five-ring components. This fluid contains no additives. Typical properties for these fluids are presented in table I.

APPARATUS

The ball-on-disk sliding-friction apparatus is shown in figure 1. The test specimens were contained inside a stainless-steel chamber. The atmosphere was controlled with respect to oxygen and moisture content.

A stationary 0.476-centimeter-radius ball was placed in sliding contact with a rotating 6.3-centimeter-diameter disk. A sliding speed of 17 meters per minute (100 rpm) was maintained. A normal load of 1 kilogram (initial Hertz stress of $10^9 \text{ N}/\text{m}^2$) was applied with a deadweight. Balls and disks were made of CVM M-50 steel having a Rockwell C hardness of 62 to 64.

The disk was partially submerged in a polyimide cup containing the test lubricant and was heated by induction. Bulk lubricant temperature was measured with a thermocouple. Disk temperature was monitored with an infrared pyrometer. Frictional force was measured with a strain gage and was recorded on a strip-chart recorder.

Several atmospheres, with oxygen concentrations from 20 percent (air) to 0.001 percent (nitrogen), were used in this study. The intermediate oxygen concentrations studied were 10, 5, 1, 0.1, and 0.01 percent. These intermediate oxygen levels were obtained by mixing air and nitrogen streams. The moisture content of all atmospheres was less than 50 parts per million.

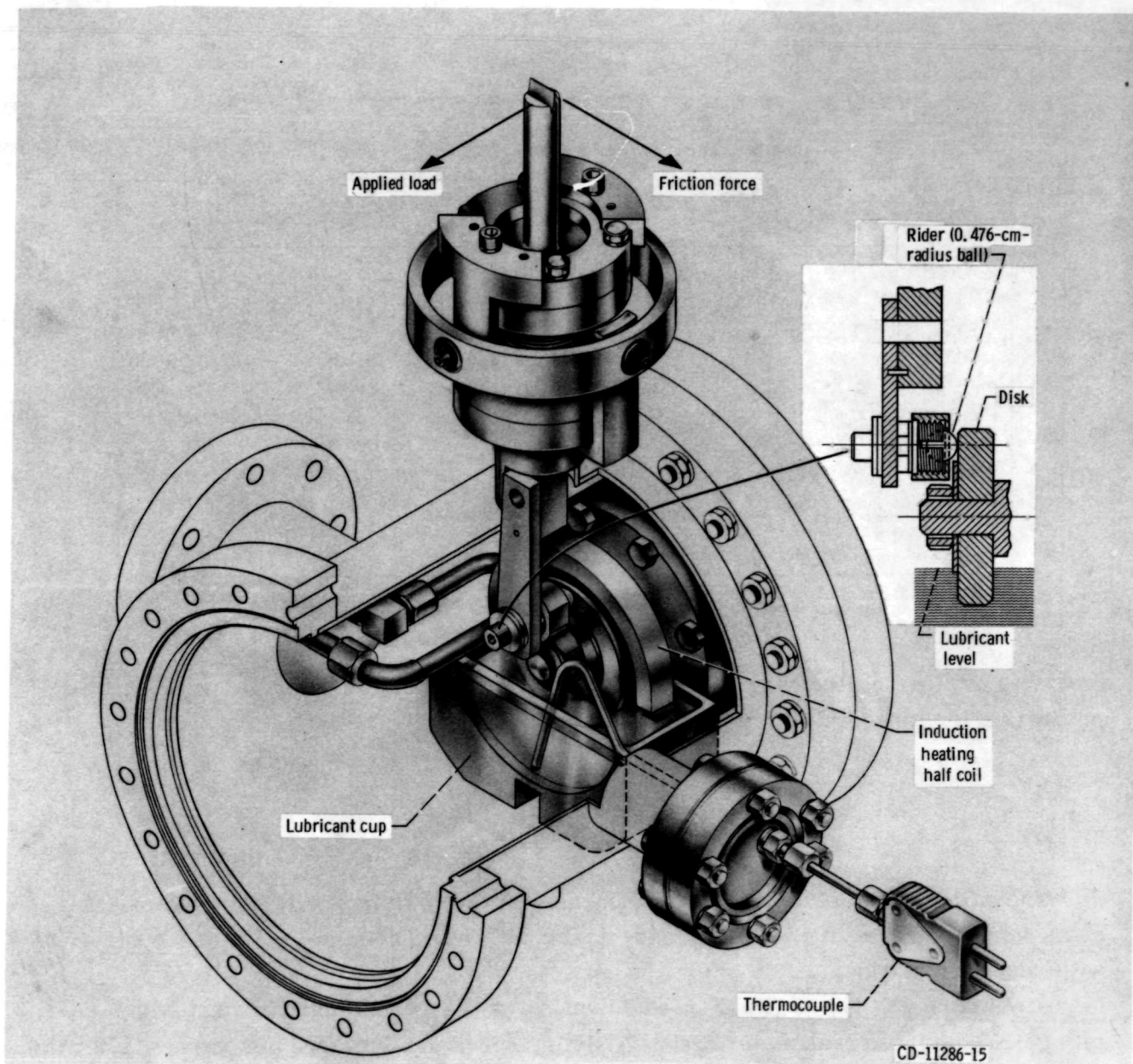


Figure 1

Oxygen concentration in the test atmosphere was monitored by an oxygen analyzer with an accuracy of approximately ± 10 percent of the true value. Moisture content was monitored by a moisture analyzer with an accuracy of ± 10 parts per million.

PROCEDURE

Disks were ground and lapped to a surface finish of 10×10^{-8} meter ($4 \mu\text{in.}$) CLA and balls to 2.5×10^{-8} meter ($1 \mu\text{in.}$) CLA. Specimens were scrubbed with a paste of

levigated alumina and water, rinsed with tap water and distilled water, then placed in a desiccator.

The C-ether used in this study was degassed at approximately 150° C (302° F) under a vacuum (2 torr) for 1 hour. Measurements by means of the Karl Fischer technique indicate that this procedure reduces dissolved water content in C-ethers to less than 20 parts per million.

The specimens were assembled, and 7.0×10^{-5} cubic meter (70 ml) of lubricant was placed in the lubricant cup. The test chamber (3.7×10^{-3} cubic meter volume) was purged with the test atmosphere for 10 minutes at a flow rate in excess of 5×10^{-2} cubic meter per hour. The disk was heated by induction to test temperature while it was rotating, and the ball was then loaded against the disk. Test-atmosphere flow rate was reduced to 3.5×10^{-2} cubic meter per hour, and a pressure of 6.9×10^{-3} newton per square meter (1 psig) was maintained in the chamber. The lubricant was heated only by heat transfer from the rotating disk. The bulk lubricant temperature was essentially the same as the disk temperature at disk temperatures to 100° C (212° F). At disk temperatures of 200° and 300° C (392° and 572° F), the bulk lubricant temperatures stabilized at approximately 150° and 200° C (302° and 392° F), respectively.

Frictional force and bulk lubricant temperature were recorded continuously. Disk temperature was monitored continuously. Each experiment was terminated after a 25-minute duration, and the rider (ball) wear scar was measured and recorded.

RESULTS

C-Ether Wear Results

Average rider (ball) wear rate as a function of disk temperature for a number of oxygen concentrations is presented in figure 2. The results for the 20-percent-oxygen (air) atmosphere are from reference 14.

Two different wear modes are observed in figure 2. At high oxygen levels (10 to 20 percent), wear decreased with increasing temperature from 25° to 100° C (77° to 212° F) and increased with further increasing temperature from 100° to 300° C (212° to 572° F). At all other oxygen levels (0.001 to 5 percent) wear decreased with increasing temperature over the entire temperature range.

The C-ether exhibited lower wear at high oxygen levels (10 to 20 percent) from 25° to 200° C (77° to 392° F) and at low oxygen levels (0.001 to 1.0 percent) from 200° to 300° C (392° to 572° F).

Table II summarizes all of the C-ether friction and wear results.

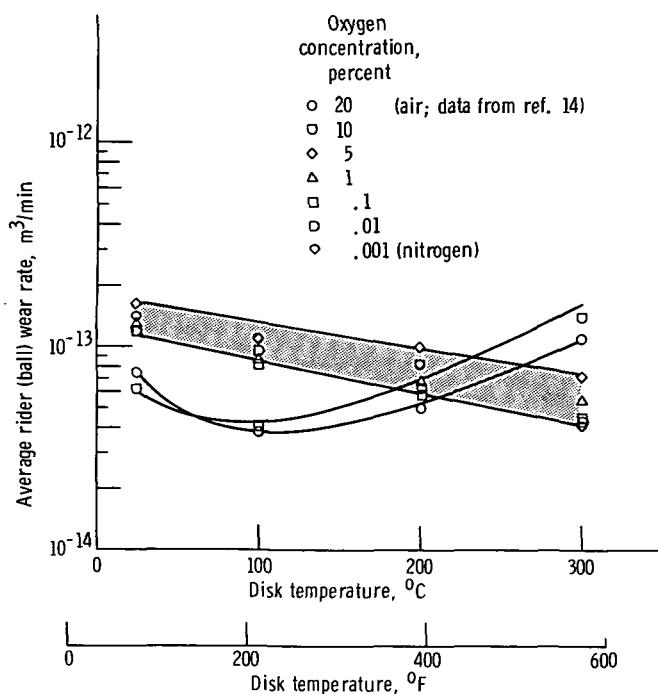


Figure 2. - Average rider (ball) wear as a function of disk temperature with a C-ether base fluid in atmospheres with several oxygen concentrations. Specimen material, CVM M-50 steel; load, 1 kilogram; sliding speed, 17 meters per minute (100 rpm); test duration, 25 minutes.

TABLE II. - SUMMARY OF FRICTION AND WEAR RESULTS FOR
C-ETHER BASE FLUID

[Test conditions: load, 1 kg; sliding speed, 17 m/min (100 rpm);
duration, 25 min; material, CVM M-50 steel.]

Disk temperature, $^{\circ}C$ ($^{\circ}F$)	Concentration of oxygen in test atmosphere, percent (by volume)						
	0.001 (nitrogen)	0.01	0.1	1.0	5.0	10	20 (air) (a)
Coefficient of friction							
25 (77)	0.08	0.07	0.07	0.07	0.08	0.10	0.12
100 (212)	.18	.18	.17	.14	.14	.14	.16
200 (392)	.21	.24	.23	.21	.20	.18	.18
300 (572)	.22	.22	.20	.17	.14	.14	.20
Average rider (ball) wear rates, m^3/min							
25 (77)	14×10^{-14}	12×10^{-14}	11×10^{-14}	12×10^{-14}	16×10^{-14}	6.2×10^{-14}	7.5×10^{-14}
100 (212)	11.0	9.4	8.3	8.6	11.0	3.9	3.8
200 (392)	8.0	6.5	5.8	6.4	10.0	8.4	5.0
300 (572)	4.2	4.2	4.4	5.4	7.6	14.0	11.0

^aData from ref. 14.

Five-Ring Polyphenyl Ether Wear Results

The unformulated polyphenyl ether (henceforth referred to herein as 5P-4E) was tested at two oxygen levels, 20 percent (air) and 0.001 percent (nitrogen). Figure 3 contains the wear results for this fluid in both atmospheres as a function of disk temperature.

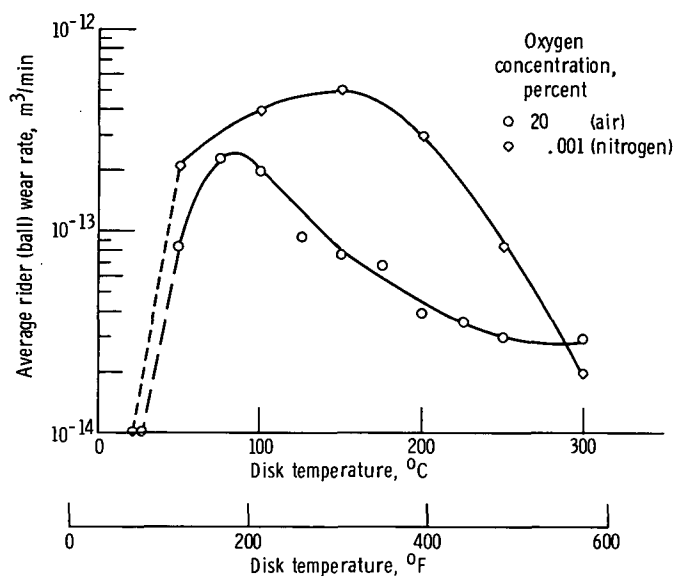


Figure 3. - Average rider (ball) wear as a function of disk temperature with a five-ring polyphenyl ether in atmospheres with two oxygen concentrations. Specimen material, CVM M-50 steel; load, 1 kilogram; sliding speed, 17 meters per minute (100 rpm); test duration, 25 minutes.

Oxygen level of 20 percent (air). - Negligible rider wear ($<10^{-14}$ m³/min) was observed at 25° C (77° F) with 5P-4E in both air and nitrogen. This is a fluid-film effect resulting from the high viscosity of 5P-4E at 25° C (77° F) and the good surface finish ($<10 \times 10^{-8}$ m (<4 μ in.) CLA) of the test specimens. Maximum wear occurred at approximately 75° C (167° F). From 75° to 300° C (167° to 572° F), rider wear decreased with increasing temperature.

Oxygen level of 0.001 percent. - Much higher wear was observed in nitrogen than in air over practically the entire temperature range. The maximum wear occurred at approximately 150° C (302° F). A rapid decrease in wear was observed from 150° to 300° C (302° to 572° F).

Comparison of Wear for C-Ether and 5P-4E

Wear rates for both C-ether and 5P-4E at oxygen levels of 20 percent (air) and 0.001 percent (nitrogen) are presented in figure 4. The C-ether yielded lower wear than the 5P-4E in dry air from 50° to 195° C (122° to 383° F) and in dry nitrogen from 50° to 265° C (122° to 509° F).

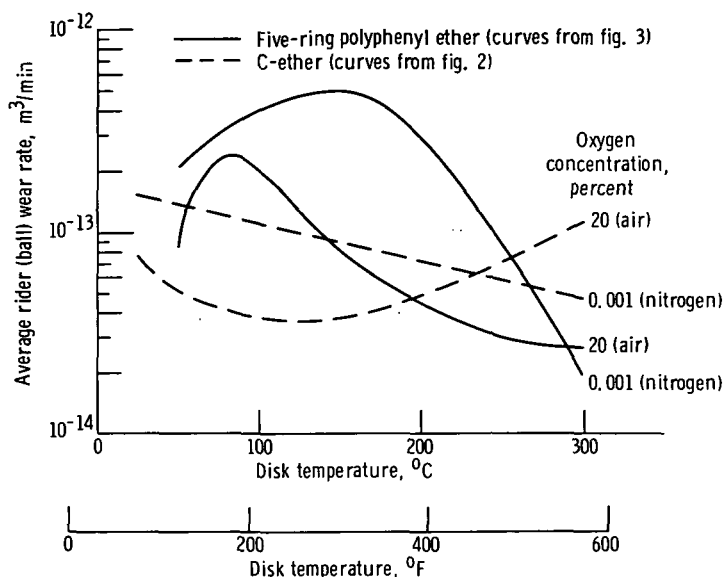


Figure 4. - Average rider (ball) wear rate as a function of disk temperature with a C-ether and a five-ring polyphenyl ether in atmospheres with two oxygen concentrations. Specimen material, CVM M-50 steel; load, 1 kilogram; sliding speed, 17 meters per minute (100 rpm); test duration, 25 minutes.

It is evident that the structural modifications of the C-ether have minimized the lower temperature (<200° C (<392° F)) boundary lubrication problems associated with the polyphenyl ethers. It should also be noted that these modifications increase wear at the higher disk temperature in both atmospheres. However, additives to the C-ether base fluid may lessen or eliminate this effect (ref. 14).

Coefficient of Friction

The coefficient of friction as a function of disk temperature for the C-ether at a number of oxygen levels is shown in figure 5. The data fall into a band that has the general trend of increasing friction with increasing disk temperature to 200° C (392° F). The coefficient of friction as a function of temperature for 5P-4E in air and in nitrogen

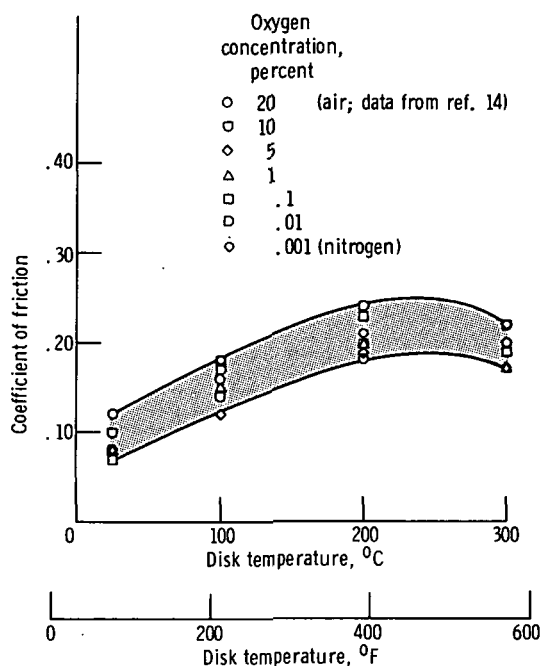


Figure 5. - Coefficient of friction as a function of disk temperature with a C-ether base fluid in atmospheres with several oxygen concentrations. Specimen material, CVM M-50 steel; load, 1 kilogram; sliding speed, 17 meters per minute (100 rpm); test duration, 25 minutes.

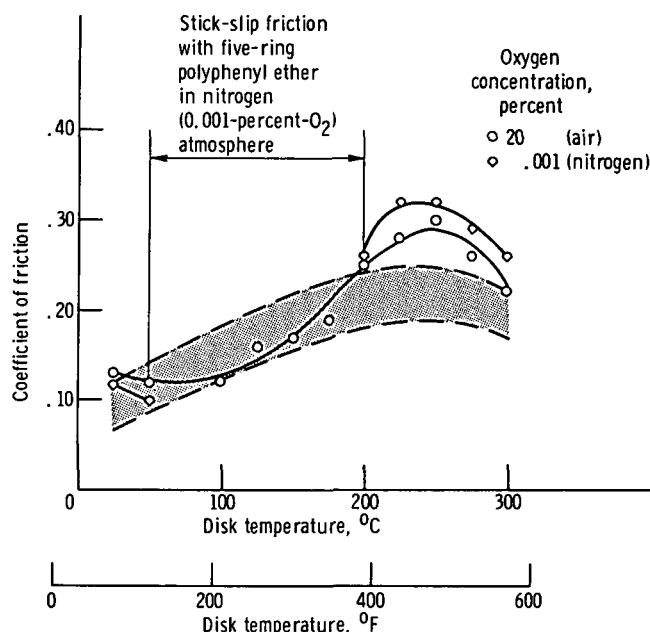


Figure 6. - Coefficient of friction as a function of disk temperature with a five-ring polyphenyl ether in atmospheres with two oxygen concentrations. Specimen material, CVM M-50 steel; load, 1 kilogram; sliding speed, 17 meters per minute (100 rpm); test duration, 25 minutes.

is given in figure 6. The friction-coefficient band obtained with the C-ether at all oxygen levels is shown for comparison.

At an oxygen level of 20 percent (air), the 5P-4E exhibits the similar trend of increasing friction with increasing temperature over most of the temperature range. At an oxygen level of 0.001 percent (nitrogen) from 50° to 200° C (122° to 392° F), the 5P-4E exhibited stick-slip, or chattering friction, which is indicative of very poor boundary lubrication. This friction region coincides with the high-wear region in figure 3. It is evident that the C-ether exhibited lower friction coefficients than the 5P-4E over the entire temperature range in nitrogen. In air, the C-ether exhibited similar or lower friction coefficients than the 5P-4E.

DISCUSSION

Highly aromatic fluids have often been reported as poor boundary lubricants in low-oxygen environments. Appeldoorn and Tao (ref. 11) showed that heavy aromatics (such as 1-methylnaphthalene) scuffed at low loads (1 kg) in dry argon in ball-on-cylinder tests. They also found that small amounts of dissolved oxygen or dissolved water prevented

scuffing with aromatic materials. Fein and Kreuz (ref. 15) reported that benzene (an aromatic) yielded high wear in four ball tests at low oxygen concentrations. The lowest wear was observed when air or oxygen was used as the saturating atmosphere. Spar and Damasco (ref. 12) studied a four-ring polyphenyl ether in nitrogen with a four-ball tester. High wear was observed, and the maximum wear occurred between 135° and 204° C (275° and 400° F). Vinogradov et al. (ref. 16) reported lower friction coefficients and higher seizure loads for benzene in air than in argon in four-ball tests. Similar phenomena have been reported with polyphenyl ethers in vane-pump studies (ref. 8) and with a ball-on-disk apparatus (ref. 10).

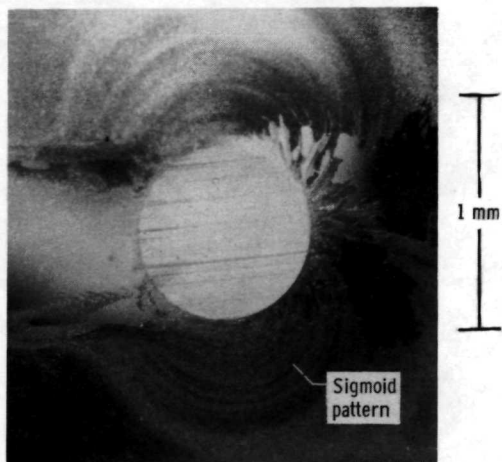
Therefore, the fact that aromatic fluids are poor boundary lubricants in the absence of oxygen and water vapor at temperatures below 200° C (392° F) is well documented. The reasons for this behavior, however, are not well understood.

A phenomenon that has been observed by many investigators in sliding-friction experiments is the formation of "friction polymer" (refs. 15, 17, 18, and 19). This is the carbonaceous polymer-like residue found in and around wear scars in sliding experiments. It has been theorized (ref. 17) that this highly viscous product of sliding actually causes the formation of a partial elasto-hydrodynamic film. This film then greatly reduces friction and wear.

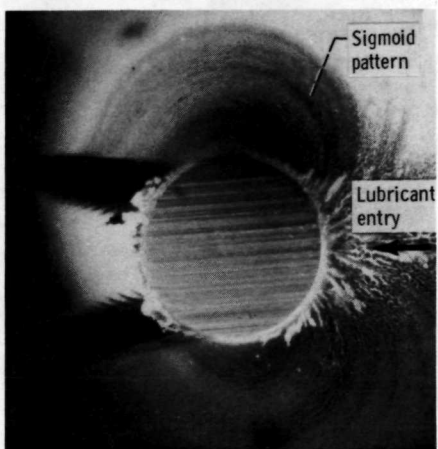
It would follow that materials that produce copious amounts of friction polymer would be good boundary lubricants. Based on the previous discussion, one could then speculate that highly aromatic fluids require high oxygen concentrations in order to produce adequate quantities of friction polymer. Hermance and Egan (ref. 18) found that in air, aromatic compounds did produce larger quantities of friction polymer than nonaromatic compounds under identical test conditions. Fein and Kreuz (ref. 15) also observed increased production of friction polymer with benzene in air.

Friction polymer was observed around most of the wear scars in this study. There were no quantitative measurements, but visual observation did not reveal any correlation between the amount of friction polymer present and the level of wear. Three typical wear scars for tests at 25° C (77° F) and at three oxygen levels are shown in figure 7. Three wear scars obtained under similar oxygen levels and at 300° C (572° F) are shown in figure 8. In figure 7, the three scars appear quite similar despite the wide range of oxygen concentration. Friction polymer can be seen in the contact inlets. "Tails" appear in the contact exits where this material and lubricant have been expelled. Sigmoid-shaped patterns are noted around the wear scars in figure 7. Recently, Schnurmann and Pedersen (ref. 20) have related these sigmoid patterns to the combined action of a magnetic field and a hydrodynamic flow field. It is theorized that the magnetic field is the result of an electrical discharge at the frictional contact.

In general, the friction polymer appeared to be held more tenaciously to the specimen surface at the lower temperatures. At the higher temperatures, an alcohol rinse to



(a) Oxygen concentration, 20 percent (air).

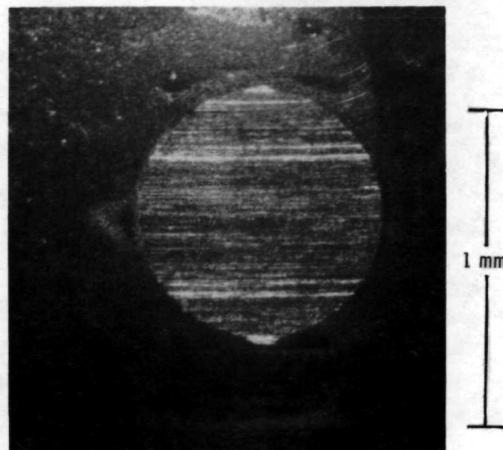


(b) Oxygen concentration, 1 percent.

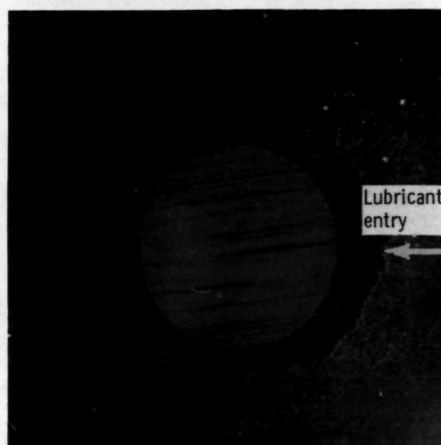


(c) Oxygen concentration, 0.001 percent (nitrogen).

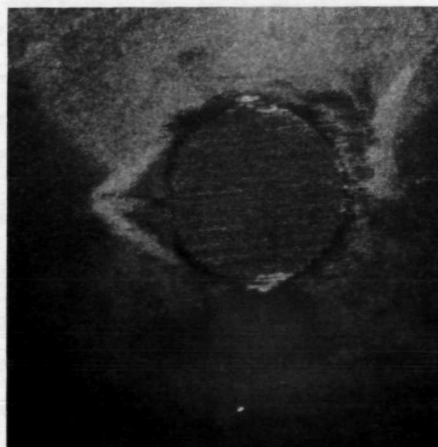
Figure 7. - Typical rider (ball) wear scars with a C-ether at a disk temperature of 25° C (77° F) in atmospheres with three oxygen concentrations. Specimen material, CVM M-50 steel; load, 1 kilogram; sliding speed, 17 meters per minute (100 rpm); test duration, 25 minutes.



(a) Oxygen concentration, 20 percent (air).



(b) Oxygen concentration, 5 percent.



(c) Oxygen concentration, 0.001 percent (nitrogen).

Figure 8. - Typical rider (ball) wear scars with a C-ether at a disk temperature of 300° C (572° F) in atmospheres with three oxygen concentrations. Specimen material, CVM M-50 steel; load, 1 kilogram; sliding speed, 17 meters per minute (100 rpm); test duration, 25 minutes.

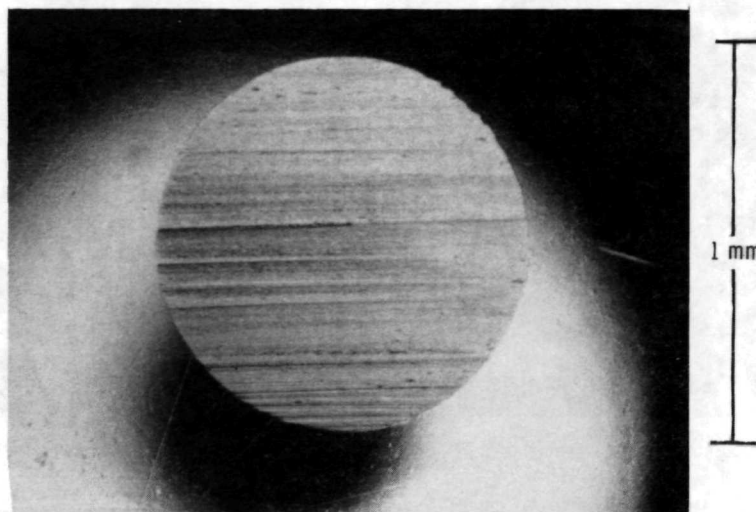


Figure 9. - Cleaned rider wear scar with a five-ring polyphenyl ether at a disk temperature of 200°C (392°F) in an atmosphere with an oxygen concentration of 0.001 percent (nitrogen). Specimen material, CVM M-50 steel; load, 1 kilogram; sliding speed, 17 meters per minute (100 rpm); test duration, 25 minutes.

remove residual oil easily detached the debris around the wear scar.

A cleaned wear scar obtained with 5P-4E in dry nitrogen in the high-wear regime is shown in figure 9. Although the chattering friction observed during this test is indicative of a scuffing type of wear, the wear scar is surprisingly smooth. This type of scuffing wear was also observed by Appeldoorn and Tao (ref. 11).

The apparent lack of any correlation between the amount of observed friction polymer and the level of wear may be the result of a number of factors. First, it is obviously somewhat difficult to visually compare amounts of friction polymer around the wear scars. Secondly, compositional changes in the friction polymer due to changes in test conditions (i. e., changes in temperature or oxygen concentration) may have a greater effect on the wear process than the variation in the quantity generated. Finally, as mentioned before, friction polymer appeared to be more easily detached from the specimen surface after tests at high temperatures. Solubilization by the bulk oil at the higher temperatures is a distinct possibility. Obviously, there is a need for more-controlled experiments to measure exact amounts and compositions of generated friction polymers.

Recently, Goldblatt (ref. 21) has put forth a theory which may explain the anomalous behavior of aromatic compounds in friction and wear tests. His theory involves the formation of a radical anion from an adsorbed polynuclear aromatic compound. This anion could then undergo several reactions depending on the conditions. In a dry inert atmosphere (nitroben or argon), it could react directly with the metal surface to

produce a type of corrosive wear. It would react with paraffinic material (if present) to produce friction polymer (called surface resin by Goldblatt). Finally, if either water or oxygen is present, a type of quenching reaction could take place which would eventually lead to the production of friction polymer. These reactions would then explain why aromatics are poor boundary lubricants under inert conditions and are improved by the presence of water, oxygen, or paraffins.

However, it must be stated that many other factors such as fluid wettability, specimen metallurgy, and the presence of catalytic agents may play important roles in the boundary lubrication by aromatic lubricants.

SUMMARY OF RESULTS

The effect of various oxygen concentrations, ranging from 20 percent (air) to 0.001 percent (nitrogen), on the boundary-lubricating characteristics of an unformulated C-ether was studied with the use of a ball-on-disk sliding-friction apparatus. Disk temperatures ranged from 25° to 300° C (77° to 572° F). Other conditions included a load of 1 kilogram (initial Hertz stress of 10^9 N/m²), a surface speed of 17 meters per minute (100 rpm), and a test duration of 25 minutes. Test specimens were made of CVM (consumable electrode vacuum melted) M-50 steel. The C-ether friction and wear results were compared with those obtained with a five-ring polyphenyl ether. The major results were the following:

1. The C-ether yielded lower wear and lower friction coefficients than a five-ring polyphenyl ether in air (20 percent O₂) and nitrogen (0.001 percent O₂) over most of the temperature range.
2. The C-ether exhibited lower wear at high oxygen levels (10 to 20 percent O₂) from 25° to 200° C (77° to 392° F) and at low oxygen levels (0.001 to 1.0 percent O₂) from 200° to 300° C (392° to 572° F).
3. Friction polymer was observed around the wear scars of most test specimens. Friction-polymer generation and its effect on wear are discussed in light of current theories.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, October 27, 1972,
501-24.

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